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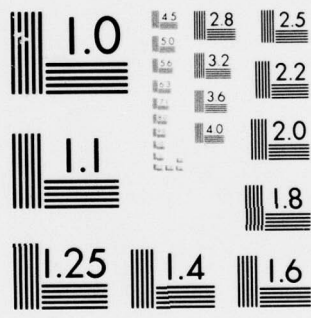
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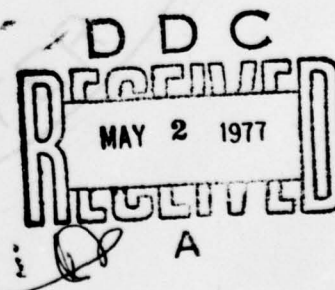
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EVALUATION OF POLYPHENYLQUINOXALINES DERIVED FROM AN INEXPENSIVE TETRAKETONE MONOMER

STANLEY E. WENTWORTH and MICHAEL J. HUMORA
POLYMER AND CHEMISTRY DIVISION

March 1977



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ABSTRACT

Polyphenylquinoxalines (PPQ's), which are superior high-temperature-resistant resins, have long been considered too costly for most aerospace applications. This is principally due to the high cost of the tetraketone monomers from which they are prepared. A recently developed alternate synthesis of these tetraketones promises to dramatically lower their cost. A PPQ has been prepared from one of these low-cost tetraketones and a series of comparisons made with the analogous PPQ derived from the conventionally prepared tetraketone. It is concluded that the PPQ derived from the tetraketone prepared via this new route is essentially equivalent to the PPQ obtained from the original monomer. Thus, the new synthesis has not introduced any impurities peculiar to it which have a deleterious effect on the resultant polymer.

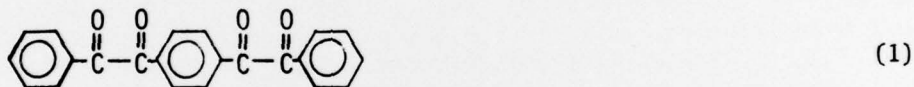
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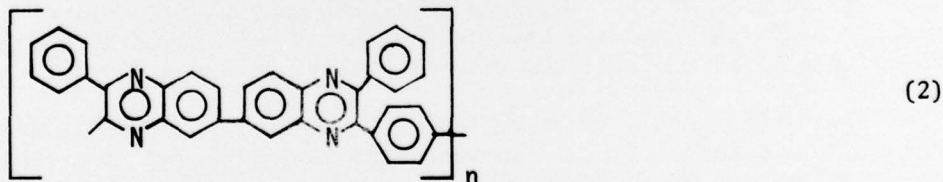
INTRODUCTION

Polyphenylquinoxalines (PPQ's) are among the very best of the currently available high-temperature-resistant resins both in terms of thermal stability and processability. A significant barrier to their widespread use as high-temperature-resistant adhesives or resin matrices for advanced composites, however, has been their prohibitively high cost. This has been largely due to the high cost (approximately \$1500 per pound) of the tetraketone monomers from which they are prepared. These tetraketones were originally synthesized in only modest yield from expensive starting materials using experimentally cumbersome reactions.¹ Although alternative tetraketone syntheses have been reported,²⁻⁵ these have not been shown to be significantly less costly than the original method. Recently, however, Kratzer et al.⁶ have described a synthesis of tetraketones based on the benzoin condensation which promises to dramatically reduce the cost (to \$15 to \$20 per pound on a large scale) of these monomers.

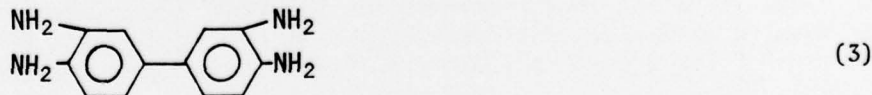
It was considered necessary to insure that tetraketones produced via this route did not contain impurities unique to the route which would have a detrimental effect on the properties of the PPQ's derived from them. Accordingly, a sample of 1,4-bis(phenylglyoxaloyl)benzene (1) prepared via this route was secured and a



PPQ (2) prepared from it by reaction with 3,3'-diaminobenzidine (3). Two different procedures reflecting laboratory and semi-pilot-plant practice were used



to prepare the polymer. These same procedures were used to prepare the same polymer from 1,4-bis(phenylglyoxaloyl)benzene synthesized via the original route for



1. AUGL, J. M., and WRASIDLO, W. J. *Synthesis and Evaluation of New Phenylated Polyquinoxalines*. Naval Ordnance Laboratory NOLTR 69-120, October 1969.
2. HIGGINS, J., and JONES, J. F. *The Preparation of Aromatic α -Diketones*. Transactions of the Illinois Academy of Science, v. 64, 1971, p. 97-98.
3. NEILANDS, O., and KREIGBERGA, J. *Method for the Preparation of Aromatic α -Diketones*. German Patent 2,225,459, December 13, 1973.
4. WENTWORTH, S. E. *Preparation of 1,4-bis(Phenylglyoxaloyl)benzene*. U. S. Patent 3,829,497, August 13, 1974.
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6. KRATZER, R. H., PACIOREK, K. L., and KARLE, D. W. *Modified Benzoin Condensation of Terephthaldehyde with Benzaldehyde*. Journal of Organic Chemistry, v. 41, 1976, p. 2230.

comparison. A number of pertinent properties were then determined for each of the four polymers. The results are given below.

EXPERIMENTAL

Materials

The 3,3-diaminobenzidine was obtained from Whittaker Corp., San Diego, California, and recrystallized according to the procedure of Hergenrother⁷ before use. The 1,4-bis(phenylglyoxaloyl)benzene was obtained from Whittaker Corp., (original route) or Ultrasystems Inc., Irvine, California (new route - material supplied through the courtesy of K. L. Paciorek) and used as received or recrystallized from ethanol before use. The m-cresol was vacuum distilled before use. Technical xylene was used as received.

Polymer Synthesis

A. Laboratory Practice. Into a 50-ml one-neck flask fitted with a magnetic stirrer was placed a solution of 0.2143 g (0.001 mole) of 3,3'-diaminobenzidine in 10.0 ml m-cresol. An additional 2.0 ml of m-cresol was used to aid in quantitative transfer. To this was added in one portion with stirring a solution of 0.3424 g (0.001 mole) of recrystallized 1,4-bis(phenylglyoxaloyl)benzene in 10.0 ml m-cresol. Again, 2.0 ml m-cresol was used to aid in transfer. Finally, the addition funnel was rinsed with 1 ml of m-cresol. The reaction mixture was warmed to 100 C over 1 hour by means of an oil bath. After 23 hours at 100 C the viscous dark amber solution was removed from the bath and allowed to cool to ambient temperature. The polymer was isolated by pouring into methanol and collecting the resultant precipitate on a glass-fritted funnel. After washing with several portions of hot methanol, the polymer was air dried overnight, finely divided, and dried in vacuo at 85 C for 6 hours to yield the solvent-free polymer for further study.

B. Semi-Pilot-Plant Practice. Using essentially the procedure of Hergenrother,⁷ 1.2518 g (0.0058 mole) of 3,3'-diaminobenzidine was placed in a 100-ml one-neck flask fitted with a magnetic stirrer followed by 20 ml of 1:1 m-cresol/xylene. To the stirred slurry was added 2.000 g (0.0058 mole) of as-received 1,4-bis(phenylglyoxaloyl)benzene over a 4-minute period during which time the temperature was maintained at 20 C by means of a water bath. After stirring at ambient temperature for 24 hours, the extremely viscous dark amber solution was poured into an excess of methanol resulting in a filamentous precipitate which was isolated by filtration. After washing with five 40 ml portions of methanol, the polymer was dried in vacuo at 50 C for 2 days to yield the threadlike polymer for further study.

Characterization and Thermal Analysis

Infrared spectra were obtained with a Perkin-Elmer Model 137 infrared spectrophotometer. Viscometry was carried out in a Cannon-Ubbelohde dilution viscometer using m-cresol at 30 C as the solvent. Thermogravimetric analyses (TGA) were performed with a Du Pont Model 951 Thermogravimetric Analyzer using a heating rate

7. HERGENROTHER, P. M. *Poly(phenylquinoxalines)*. *Macromolecular Syntheses*, v. 5, 1974, p. 17.

of 10 C per minute in an atmosphere of static air. Isothermal aging studies were conducted with this equipment in a static air environment. Torsional braid analyses were performed with a Chemical Instruments Corporation Model 100-B1 Torsional Braid Analyzer. A controlled heating rate of 4.2 C per minute was provided by a Tetrahedron Associates Model ATC-200 Temperature Indicator Controller in conjunction with a Research Incorporated Model 5310 Data Track card reader. All determinations were conducted in a nitrogen atmosphere with 8" multifilament quartz braids.

RESULTS AND DISCUSSIONS

A summary of the pertinent properties determined for each of the PPQ's is given in the table and discussed in detail below.

COMPARISON OF PPQ's DERIVED FROM ORIGINAL
AND NEW TETRAKETONE SYNTHESSES

Property	Laboratory		Semi-Pilot-Plant	
	Original	New	Original	New
Yield, %	96.2	97.3	95.0	96.1
Intrinsic Viscosity, dl/g	1.22	0.73	1.65	1.76
Polymer Decomp. Temp, deg C	597	595	630	625
Half-Life,* Hr	84	45-1/2	62-1/2	70.0
T _g , deg C†	356	353	360	363

*Static air - 400 C

†By torsional braid analysis

Yield

The yield in a polycondensation reaction is highly dependent upon monomer purity and is thus a sensitive indicator of that property. In this study, all four preparations gave very high yields which deviated from 100%, almost surely solely as a result of mechanical loss in the isolation procedure. Thus, this criterion shows the two monomers to be identical.

Infrared Spectra

Infrared spectra of thin films cast from chloroform solution were taken of the two polymers prepared using semi-pilot-plant conditions. They are identical in all respects, again indicating the identity of the two monomers.

Intrinsic Viscosity

Just as with yield, molecular weight is highly dependent on monomer purity. Thus, intrinsic viscosity, which is a function of molecular weight, is another sensitive indicator of this property. In the present study, two sets of results have been obtained. Using semi-pilot-plant conditions, both monomers yield a polymer of very high intrinsic viscosity while under laboratory conditions the viscosities are somewhat lower and show greater deviation. Since variations of this sort occur even with two consecutive polymerizations with the same batch of monomer (probably due to small weighing or transfer errors) and since even the lower viscosity represents a high polymer, these results are not considered to indicate a significant difference in the two monomers. The results from the semi-pilot-plant runs strongly support this conclusion.

Polymer Decomposition Temperature

This parameter is not so much a quantitative measure of monomer purity as it is of the presence of low levels of materials leading to polymer defects which result in diminished thermal stability. As can be seen, no significant difference in decomposition temperature is observed which can be attributed to the source of the monomer, though there does appear to be a molecular weight dependence, the higher viscosity semi-pilot-plant polymers having the higher decomposition temperatures.

Half-Life

Half-life in air at elevated temperature (400 C in the present case) is a much more sensitive indicator of the presence of polymer defects or structural factors leading to thermal oxidative instability than is polymer decomposition temperature which is determined by rapidly scanning to very high temperature. The long residence time at elevated temperature in the half-life experiment permits a discrimination of thermal oxidative susceptibility which may derive from only a small difference in the concentration of thermally labile structural features. Such a difference is easily missed in the TGA experiment. By the same token, half-life will be highly dependent on sample physical state, a finely ground sample with a high surface-to-volume ratio having a shorter half-life than a less finely ground one. Both of these factors are thought to be operating in the present study. The seemingly dramatic drop in half-life of the polymer prepared under laboratory conditions from the new monomer relative to that of the polymer from the old monomer may be partly due to a difference in physical state. In addition, the low molecular weight of this material means that it has a greater number of end groups. Since Augl⁸ has shown that a PPQ with amino end groups has inferior isothermal aging properties, it is reasonable to attribute a substantial portion of the diminished half-life to this factor. Given the vagaries of sample dimension, the modest difference in half-life for the two semi-pilot-plant polymers is not thought to be significant, especially since it is in the opposite direction from that of the laboratory polymer. In summary, the somewhat contradictory results obtained in the isothermal aging experiments are not felt to derive from important differences in the tetraketone monomers.

8. AUGL, J. M. *Thermal Degradation of a Polyphenylquinoxaline in Air and Vacuum*. Journal of Polymer Science, Part A-1, v. 10, 1972, p. 2403.

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Glass Transition Temperature

The differences between the glass transition temperatures are probably just barely significant and seem to reflect only the differences in molecular weight, a phenomenon previously observed with PPQ's by Hergenrother.⁹ They certainly do not indicate any difference in the monomers.

CONCLUSIONS

It is obvious from the findings presented above that the tetraketone monomer prepared via the low cost benzoin condensation route contains no impurities which have a deleterious effect on the properties of the resultant PPQ's. These resins may be used with good confidence that they will perform as expected by extrapolation from resins derived from the original monomer. Thus, this new route represents a significant breakthrough in the effort to make PPQ's less costly and hence more attractive for a wide range of aerospace applications.

ACKNOWLEDGMENT

The authors gratefully acknowledge the many helpful discussions with Dr. G. Hagnauer of these laboratories.

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